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TECHNICAL REPORT

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THE FLASH EVAPORATION OF POWDERED INDIUM ANTIMONIDE

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S U M M A R Y

It has been shown experimentally for deposition rates of
practical interest, that the flash evaporation of powdered
indium antimonide from a black body source yields a stoichio-
metric vapour over a wide angular range about the axis of the
source. Films deposited by this means have an excellent
surface finish and are free from the surface defects often
associated with flash evaporation employing other source
geometries.

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1. INTRODUCTION

The preparation of high quality III-V compound films by vacuum deposition, is hampered by the relatively high vapour pressure of the group V elements. As the compound is heated, the more volatile component tends to evaporate first, producing a film with a layered structure. It may also happen that the components are distributed spatially about the source axis, each according to a different law. When this is so, the atomic composition of the film will vary with position on the substrate. Finally the vacuum deposition of III-V compounds often produces films with irregular surface texture, unsuited to planar techniques. In this memorandum the usefulness of the technique of rapidly evaporating finely powdered material, fed a few particles at a time onto a hot strip of molybdenum, is examined in relation to overcoming these problems when producing binary films containing indium and antimony.

2. DISCUSSION OF THE FLASH EVAPORATION OF PELLETS AND POWDER

If a small piece of indium antimonide is evaporated at a very low rate, we would expect to observe the material melt and emit antimony vapour followed eventually by indium vapour(ref.1). The compound decomposes into the elements, and the higher vapour pressure component antimony diffuses through the melt to the surface, where it evaporates. A film deposited on a substrate held above this vapour source would consist of a layer of antimony covered by a layer of indium with an intermediate region containing both indium and antimony. If we now consider the evaporation of a number of small pellets one after the other, we would expect the resultant films to be made up of equal numbers of inter-leaved layers of indium and antimony. This corresponds to the case where powdered indium antimonide is fed grain at a time onto a hot vaporising source. Although this film structure is still layered, uniformity exists over a smaller scale than in the previously discussed film structure of just two layers.

If the evaporation of a powder particle commences before the previous one is completely vaporised, then the discrete nature of the interleaved layers will be lost to some extent. Provided the particles evaporating at any one instant are not too close together on the source, we would not expect any interaction of significance to this discussion. Thus it would seem that to allow a small number of pieces of powder to be on the source together at various stages of evaporation, would produce uniformity on an even smaller scale than previously considered.

So far we have considered only extremely slow vaporisation. Turning now to rapid evaporation of the powder we would expect the vapour emanating from each particle to be binary in nature and still further reduce layering in the deposited films. This effect is well known(ref.2) and occurs because the diffusion of the more volatile component from within the evaporant to the evaporating surface is insufficient to maintain its concentration at the surface, thus allowing the surface temperature of the material to rise and favour the evaporation of the less volatile component.

However, with the use of very high evaporation rates, the mean free path of the evaporated species above the source may be reduced to such an extent that the cosine law of emission ceases to hold and the molecular beam becomes partly focussed. The focussing of such a beam is not desireable for large scale thin film production because it prevents the use of simple substrate support geometries for uniform thickness of deposition on many substrates simultaneously. An equally serious objection to a focussed beam arises when the beam consists of more than one type of atom, since the atomic composition of the film is then likely to vary with position on the substrate, i.e. unless the spatial distribution for each element present in the beam is of the same form as all others, the composition of the film will vary with substrate position and of course will in general be different from the composition of the material being evaporated.

The effect then, of rapidly evaporating small pellets of a compound will be to produce films with a degree of homogeneity in the direction of their thickness, but lacking in uniformity across the substrate. To study this effect, a flat extended substrate surface was held as in figure 1 above the source, normal to its axis and the variations of the thickness of the deposit over the area of the substrate was used to determine the spatial distribution of the vapour from the source. Indium antimonide, antimony or indium pellets were evaporated in this way and the film thickness determined as a function of distance from the boat axis. A standard Talysurf thickness measuring procedure was used for most of these measurements with the exception of those carried out on soft indium deposits, which required the use of a specially developed technique employing a hard silicon monoxide protective layer over the soft film(ref.3). Figure 2 shows the thickness profile of a deposit obtained from an indium antimonide pellet. It can be seen there that the film thickness on the boat axis is generally much greater than predicted by the Knudsen cosine law. The figure shows three experimental curves for which the deposition time varies from 4 s to 164 s. Since the same pellet mass was used in each case the longer deposition time corresponds to a low deposition rate and hence a low vapour pressure above the boat while the rapid deposition would be accompanied by a higher vapour pressure over the melt. The trend towards a cosine distribution as the vapour pressure is decreased (i.e. as the mean free path increases) is the sort of behaviour which one would expect. Where indium alone, rather than indium antimonide was evaporated, there was again evidence for the vapour being concentrated along the source axis. This is shown for indium in figure 3 where the extent of the focussing is even more pronounced. Somewhat surprisingly antimony with its high vapour pressure behaves quite differently as seen in figure 4, where the antimony distribution fits the cosine law very closely, even though the deposition rate was high.

It is interesting to consider the significance of these results, particularly in relation to the quality of films reported by earlier workers. Forward(ref.4) reported electron mobilities of 2 m^2/Vs for films obtained by rapidly evaporating pellets and allowing a film to condense onto a substrate heated in a tubular oven. The layer so deposited was then annealed to improve film quality. From what we have seen in figures 2 to 4 it is surprising to find that this technique may be used to produce high quality films of indium antimonide, because to have high mobilities and Hall coefficients it has always been found that the material must be stoichiometric; a very improbable event on evidence so far considered.

However when the effect of the tube oven is taken into account the results obtained by Forward are more readily understood. Using the apparatus of figure 5, small quantities of indium, antimony or indium antimonide were rapidly evaporated, and the diameter of the deposits measured for various tube oven temperatures. With the substrate held as shown, the deposit was formed in two ways, firstly by those atoms which were able to pass through the oven without striking the walls and secondly by atoms re-emitted from the inside surfaces of the oven. The former component of the deposit is limited in size by the geometry of the arrangement to a diameter d, while atoms re-emitted from the oven walls may condense on the substrate in positions outside that circle. With the oven at room temperature all deposits were of diameter d (see figure 6). For indium, oven temperatures up to 380°C did not influence the diameter of the deposit, while for antimony the deposit was much larger and the edge very poorly defined at this temperature. It appears that the sticking coefficient of indium is close to unity on the tube surface for temperatures between 20°C and 380°C and this results in an optically defined deposit of diameter d. This is also true for antimony when the oven is cold, but at 380°C antimony is re-emitted from the tube surface and able to produce a deposit of greater diameter. It is suggested that in the work of Forward, where the substrate was held in the middle of the oven, the indium rich deposit expected from consideration of the spatial distribution of indium and antimony, is modified by extra antimony coming from the tube oven wall.

This picture is complicated by considering the sticking factor of the substrate, which may itself re-emit antimony, but on this point our experiments shown in figures 5 and 6 can contribute further.   If indium antimonide is rapidly evaporated while the tube oven is at or near 380°C, the diameter of the deposit varies widely and is seen to be very sensitive to the temperature of the tube.   (This only occurs at approximately 380°C).   The presence of indium in the deposit evidently inhibits the re-evaporation of the antimony from the tube since at 380°C the deposits of the compound are of smaller diameter than the antimony deposits, (see figure 6).   A glass substrate held centrally in the tube would assume a lower temperature than the tube wall.   Thus, the indium rich, cooler deposit on the glass substrate, would re-evaporate antimony to a lesser extent than the film on the oven wall, providing a mechanism by which stoichio-metry could be achieved for the film, that is, the substrate may receive the required amount of antimony from the oven walls if the temperatures are favourable.   In this context it is interesting to note that Forward reported his highest quality films using an oven temperature of 380°C.

Finally, it is apparent that for films deposited in this way to compensate a deposit for a 30% deficiency in antimony content say, would require a slightly different oven temperature than that needed to compensate for a 20% deficiency. However the precise extent of the expected deficiency is neither predictable nor reproducible since during rapid evaporation of a pellet the molten globule moves erratically in the boat, shifting the location and direction of the effective boat axis and so influencing the thickness and composition of the film formed on the stationary substrate.   This method is therefore unsatisfactory because the film composition relies on the chance balancing of relatively large influences, the magnitudes of which are not known in advance but rather measured after the event.

The rapid evaporation of very small particles of material offers possible solutions to some of these problems, while allowing a simple and convenient means of mixing materials to achieve the particular composition required for a binary or even a ternary compound.   The use of flash evaporation for powdered compounds may enable layering to be virtually eliminated, the cosine distribution to be maintained, and film uniformity in both thickness and composition to be achieved for a number of simultaneously coated substrates.

3.   APPARATUS FOR THE EVAPORATION OF POWDER

The experiments to evaluate this evaporation technique were carried out with the apparatus shown in figure 7.   A steady controlled flow of powder was obtained from this feed arrangement.   With the cylinder stationary a limiting cone of powder forms between the hopper and the cylinder.   As the cylinder rotates, powder drops into a chute or a collimating tube which guides it to the hot vaporising source.   The speed of rotation of the cylinder determines the evaporation rate, up to a value set by the maximum rate of flow of powder from the hopper.   A vibrator serves to fluidise the powder in the hopper and ensure flow down the collimator.

With a drum of approximately 2.1 cm diameter it was found that rotation frequencies between 1.4 r/min and 8 r/min achieved the desired mass feed rates. To eliminate electrical contacts within the vacuum chamber the vibrator was driven with an a.c. signal of between 25 and 50 Hz in frequency and adjusted in amplitude to achieve a reliable powder flow.   The powder transport mechanism is capable of producing even very low feed rates, with an acceptable uniformity in the stream of particles arriving at the boat.   Rates of 1 mgm/s may be obtained, corresponding to a deposition rate of approximately 10 Å/s at the source to substrate distance of 18 cm used for these experiments.

During the initial experiments, difficulty was experienced with powder jamming in the hopper.   It was often observed that after several minutes use, the rate of flow decreased steadily until the powder jammed in the hopper and the experiment had to be terminated.   With the mechanism dismantled it was then

quite difficult to clear the capillary with a fine piece of wire, so solidly was
the powder compacted.   The powder used consisted of two fractions with typical
particle sizes of 10 μm and 150 μm.   When the smaller particles were removed by
sieving, the tendency to jam was significantly reduced and the flow rate was
extremely constant, even from run to run, provided the hopper to roller spacing
was not altered.   When a crystal oscillator film thickness monitor was used to
measure the rate of deposition, excellent control of film growth rate was
achieved.
    Problems associated with guiding the powder to the hot evaporation source have
been dealt with in separate technical memoranda(ref.5,6).   These problems are,
firstly the tendency of a collimator tube to be blocked at its exit by the
evaporant vapour, and secondly the evaporant vapour deflects some of the powder
from its path towards the source, causing powder wastage and irregular lumpy
film surfaces due to powder particles hitting and substrate.   A satisfactory
solution to these problems is to use a long glass collimating tube (say 10 inches
in length) with a large bore of perhaps 1 mm.

4.   RESULTS OF POWDER EVAPORATION

    Let us now examine the usefulness of flash evaporation of powdered indium
antimonide for preparing high quality films.   Powder was rapidly evaporated by
allowing it to fall on a heated molybdenum strip.   Glass microscope cover slips
were used as substrates because they contained sufficient calcium to enable the
films to be analysed by the x-ray fluorescence technique developed as part of
this work and described elsewhere(ref.7).   These films, when compared with films
deposited from pellets, are different in a number of ways.   Firstly the oven
grown films have a matt surface whereas the powder films are optically highly
specular.   Secondly the films produced from pellets have a regular and clean
surface appearance, even when examined under the microscope.   The flash
evaporated films although very shiny and mirror like, are 'dusty' with small
pieces of powder adhering to the surface.   A third marked difference is their
behaviour in concentrated HF.*   Oven grown films dissolve rapidly (i.e. in less
than one second) whereas the powder films are not noticeably affected after 10 s
of exposure.   This is particularly interesting in view of the known behaviour
of indium, antimony and indium antimonide in HF.   Only indium antimonide
remains little affected, indium and antimony both reacting very rapidly.   This
evidence suggests that the powder films are chemically similar to the compound
whereas for oven grown films there is sufficient unrecombined indium and
antimony to allow the films to be etched from the substrate in a very short time.
Finally the two types of films produce entirely different Debye Scherrer x-ray
patterns.   Oven grown films yield rings characteristic of small crystals of
indium antimonide with signs of either indium or antimony crystallites or both.
No diffraction rings resulted from the powder films, indicating an extremely
small crystallite size or an amorphous structure.
    A number of substrates were held in positions such that they were part of an
18 cm diameter sphere which included the source.   The significance of the
sphere for a Knudsen evaporator is that the deposit has everywhere the same
thickness, it therefore affords a simple means for checking the flux distribution
for both elements.   In Table 1 the partial thicnesses for indium and antimony
around a great circle of the substrate holder (see figure 8) are seen to be
relatively independent of the angle from the source axis.   This suggests that
both elements have a distribution which is close to the cosine law.   In figure
3, which shows the spatial disbtribution of indium when rapidly evaporated from
a small pellet, we can see that for an angle ϕ of 17° from the boat axis,

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*   This became apparent because we wanted to grow an indium oxide layer on all
    films prior to recrystallising them under a scanned electron beam and the
    process chosen for oxide growth involved the use of hydrofluoric acid.

(1.52 cm from the centre of the deposit) the thickness is only 0.5 of its maximum value, whereas for the cosine law it would drop to 0.84 of its axial value. We can say then, that the flux density for $\phi = 17^{\circ}$, is 0.5/0.84, ie 0.59 of the value predicted from its axial value by the cosine law. For the case of the spherical holder this flux distribution would produce for film No.10, an indium thickness of 0.59 of the thickness of film No.1. The angle at the source between the axis (film No.1) and film No.10 is 17°. Inspection of the normalised partial thickness values in Table 1 shows that the measured variations of typically 3% (worst case 11.8%) are much less than this and that with respect to flux distribution the powder evaporation method achieves a marked improvement on the evaporation of pellets.

Whilst in the case of these indium antimonide films the spatial distribution for both indium and antimony closely followed the cosine law, the films were nevertheless deficient in indium, (see Table 1). The reason for this was that not all the indium did in fact evaporate from the heater strip, since after the evaporation was terminated small amounts of indium could be seen at the ends where lower temperatures occurred during operation. Partly because of this difficulty and partly because of the 'dust' found on the films it was decided to use a black body source instead of a strip heater. The evaporator geometry is shown in figure 9 and is similar to commercially available units. When the powder was fed into this source with a long curved glass collimator (described in reference 5), satisfactory operation was achieved. Prior to the use of this collimator, deflection of powder by vapour from the boat limited the operating time of the apparatus by causing blockages between the end of the collimator and the powder entry aperture of the boat. The long glass collimator achieved an improved collimation but also gave the powder more kinetic energy, enabling it to enter the boat with less deflection by the vapour.

The films deposited by this technique had very clean surfaces and appeared clean even when examined under the scanning electron microscope. The spherical substrate holder employed earlier was again used for this work, but due to the need for extra shields between the powder inlet and the vapour outlet, it was necessary to locate the substrate holder in a position where the source did not lie on the surface of the same sphere as the holder. Unfortunately this prevents a cosine source from depositing films of equal thickness and so our simple check of the cosine distribution is not available in this case. What is evident from Table 2, however is that these films are all close to stoichiometric regardless of location.

Initial experiments with this source were concerned with films of thickness less than one micron because the XRF analysis method is easier to use in this region. When the deposition of films of several microns thickness was attempted, this was prevented by clogging of the input collimator. After the collimator problem had been solved it was found that films greater in thickness than approximately one micron sometimes peeled from the substrate by the time the vacuum system had been let up to atmosphere. This problem has not been solved as yet and may require the selection of a more suitable substrate material to give greater film adhesion.

5. CONCLUSION

Vacuum evaporation of indium antimonide produces molecular beams of indium and antimony. If the vapour pressure of for example antimony, above the source, is such that the mean free path is comparable to the source dimension (the dimension of the molten pool of evaporant), one expects the cosine law of vapour emission to be invalid. For antimony the cosine law has however been shown to hold even for rapid evaporation of a small pellet from a boat. Reference to figure 4 shows that for the 27 mgm of material evaporated in 5 s the cosine law applies. Since during the evaporation the antimony was not in thermal equilibrium with the boat we may estimate from vapour pressure curves and the Langmuir equation for the evaporation rate of a material, an average vapour pressure of 3 Torr, an evaporant temperature of 900°C and thence a mean free path under these conditions of 27 μm, assuming a pellet size of 3 mm diameter

and noting from Holland(ref.8) that the vapour is composed cheifly of the dimer
Sb_2. It is remarkable that the mean free path is two orders smaller than the
source dimension and yet the cosine law does not break down.

In the case of indium evaporated from a small pellet, a similar procedure
yields from the data of figure 3 a temperature of $1450^{o}C$, a vapour pressure of
6 Torr and a mean free path for the monomer In of 62 μm. This dimension is
also very small compared with the source, but in this case the cosine law is
experimentally seen to fail.

The experimental work described earlier highlights the need, when working with
molecular beams, to take into account the reevaporation of species from surfaces
within the vacuum chamber, including the substrate itself. Precise control over
film composition requires a stoichiometric molecular beam incident on the
substrate together with unity sticking factors for the molecules on the substrate
and nearby surfaces or a method of removing unwanted excess components as the
film grows. In this connection the use of an energetic ion beam of indium or
antimony, in combination with a molecular beam having a slight excess of
antimony, would be of interest because of its potential for inducing epitaxial
growth. Because of the relatively high energy required by the surface atoms,
it would probably be possible to arrange experimental conditions to favour the
loss of the excess antimony from the growing epitaxial surface.

Rapid evaporation of powdered indium antimonide offers the possibility of.
achieving stoichiometric films over large areas of substrate. With the use of
black body evaporation sources, the films have extremely smooth and clean
surfaces, suitable for the planar technique.

An interesting effect has been observed for antimony vapour, where even when
the mean free path is much less than the dimensions of the vapour source, the
cosine law was seen to hold.

6. ACKNOWLEDGEMENTS

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TABLE 1.   THICKNESS AND COMPOSITION FOR SUBSTRATE POSITIONS RANGING 17° ABOUT THE BOAT AXIS

Film batch	Far left substrate (No.10)				No.3				Central substrate (No.1)				No.6				Far right substrate (No.16)				Source temperature (°C)
	T	%I_n	t_{In}	t_{sb}	T	%I_n	t_{In}	t_{sb}	T	%I_n	t_{In}	t_{sb}	T	%I_n	t_{In}	t_{sb}	T	%I_n	t_{In}	t_{sb}	
A016	0.91	46.0	(1.01) 0.279	(0.986) 0.348	0.92	47.4	(1.06) 0.291	(0.972) 0.343	0.91	45.2	(1.00) 0.275	(1.00) 0.353	0.87	46.8	(0.99) 0.272	(0.93) 0.328	0.85	46.3	(0.956) 0.263	(0.915) 0.323	1250
A017	0.87	48.2	(1.04) 0.281	(1.00) 0.319	0.88	47.2	(1.04) 0.278	(1.03) 0.329	0.85	47.2	(1.00) 0.268	(1.00) 0.318	0.81	47.1	(0.952) 0.255	(0.955) 0.304	0.78	47.8	(0.933) 0.250	(0.908) 0.289	1250
A018	0.88	47.4	(1.07) 0.279	(0.967) 0.328	0.90	45.6	(1.05) 0.274	(1.02) 0.347	0.87	45.0	(1.00) 0.261	(1.00) 0.339	0.82	47.7	(1.00) 0.262	(0.897) 0.304	0.80	47.3	(0.970) 0.253	(0.880) 0.299	1355

NOTE: Figures shown in brackets are values normalised to the corresponding quantity for the central film.   T is total thickness (μm), t_{In} and t_{sb} are thicknesses in mgm/cm^2.

TABLE 2.   THICKNESS AND COMPOSITION OF FILMS DEPOSITED WITH THE AID OF A BLACK BODY SOURCE

Film batch	Far left substrate (No.10)	No.3		Central substrate (No.1)		No.6		Far right substrate (No.16)		Source temperature (°C)
	T(μm)	T(μm)	%I_n	T(μm)	%I_n	T(μm)	%I_n	T(μm)	%I_n	
A051	0.30	0.47	53.8	0.52	51.0	0.62	51.1	0.66	50.5	1350
A052	0.31	0.40	50.3	0.53	48.6	0.64	49.1	0.69	48.2	1550
A053	-	0.39	50.2	0.52	51.5	0.59	49.4	0.62	50.0	1550

sectional view of the
extended flat substrate

exaggerated
thickness profile

Boat axis

vertical seperation between
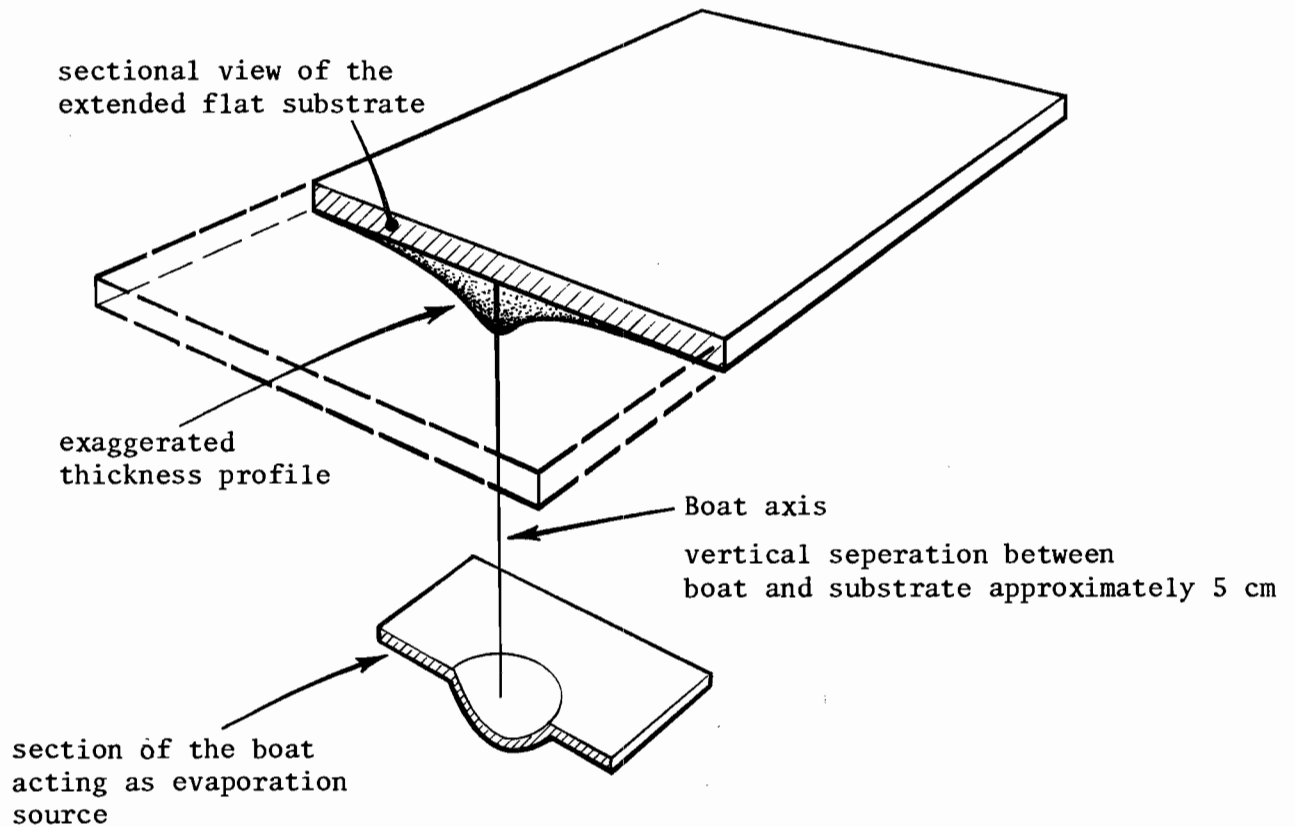boat and substrate approximately 5 cm

section of the boat
acting as evaporation
source

Figure 1.   Cross sectional view of deposition arrangement for checks on the spatial
distribution of vapour

Source to substrate
distance 5 cm pellet
mass 59 mg

KNUDSEN

4 s

7.3 s

164 s

Film thickness (μm)

Distance (cm) measured across a diameter of the substrate
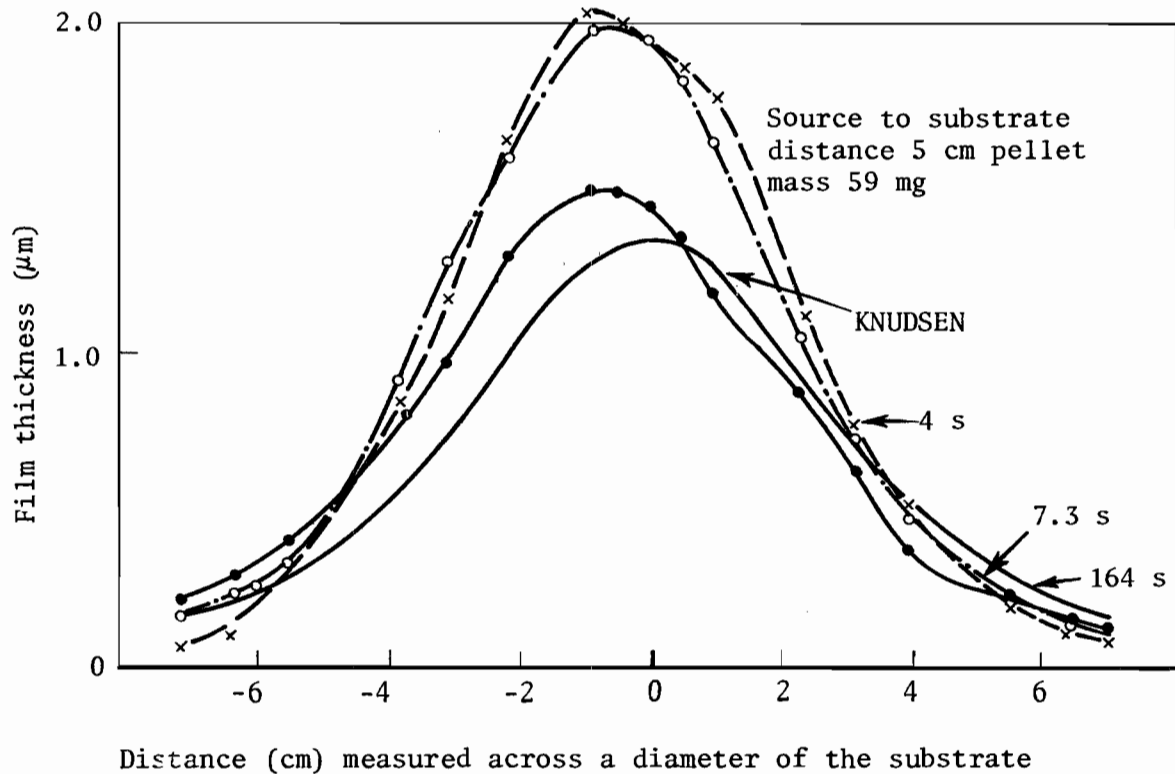
Figure 2.   Thickness profiles obtained by evaporating indium antimonide
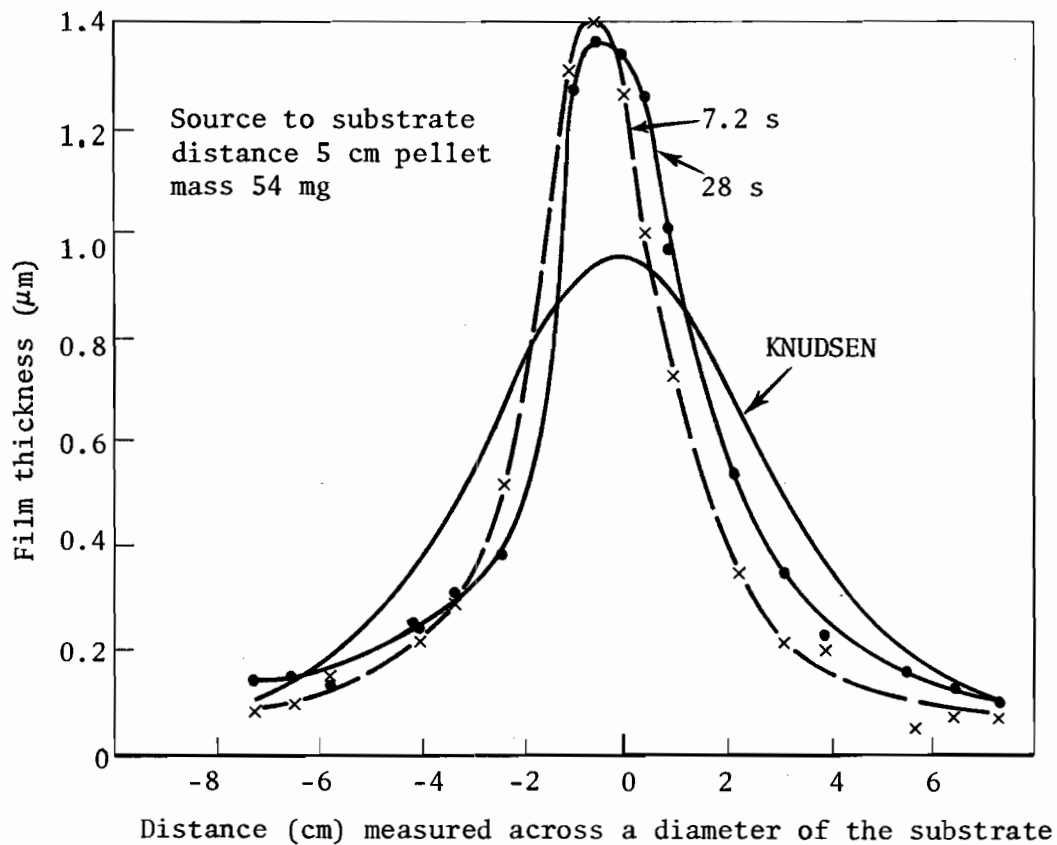pellets
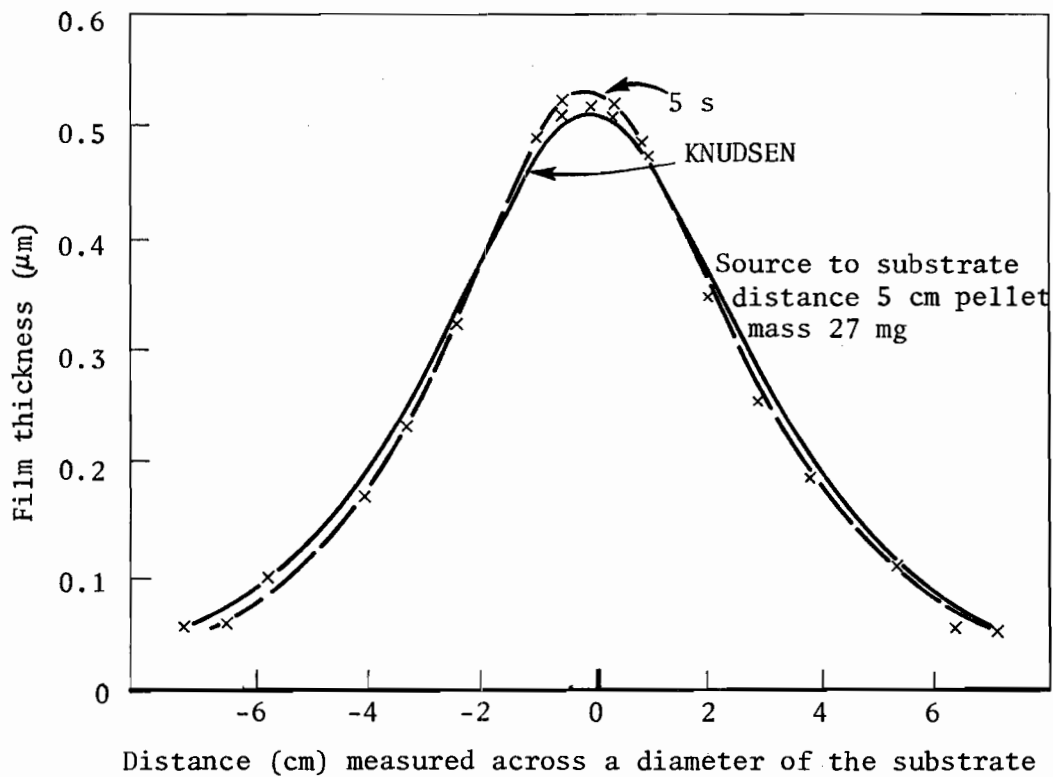
Figure 3.   Thickness profile for indium

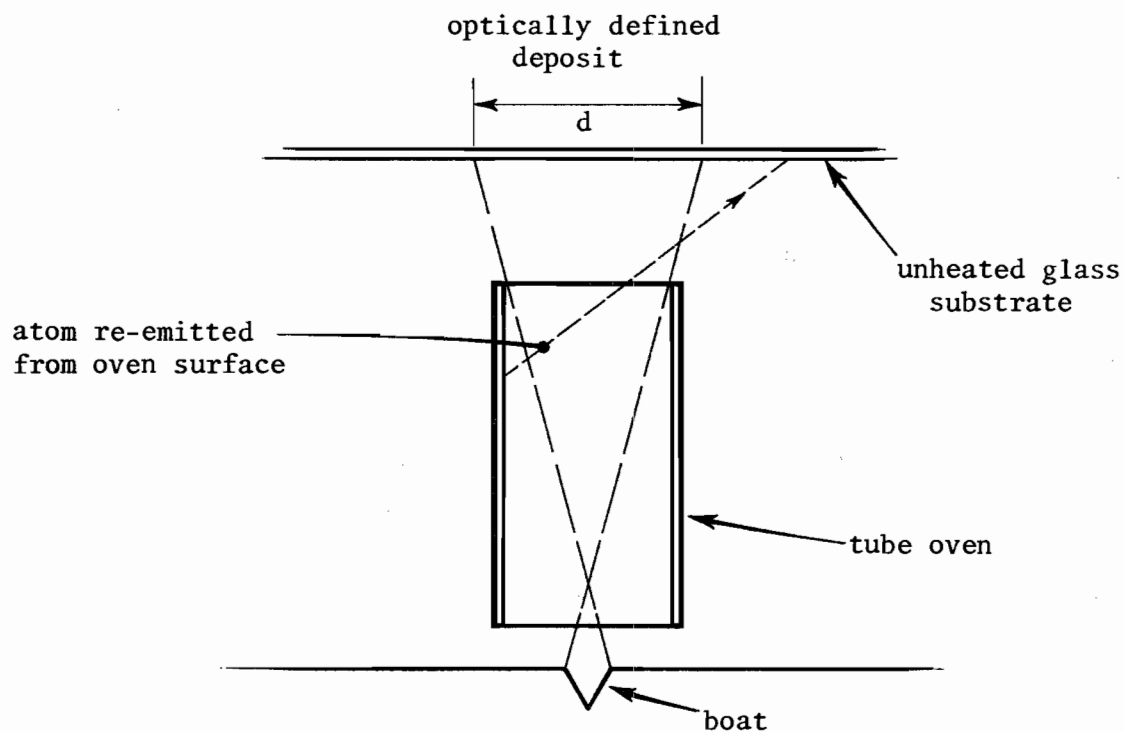Figure 4.   The spatial distribution for antimony

Figure 5. Arrangement used to observe the behaviour of indium and antimony on the oven walls
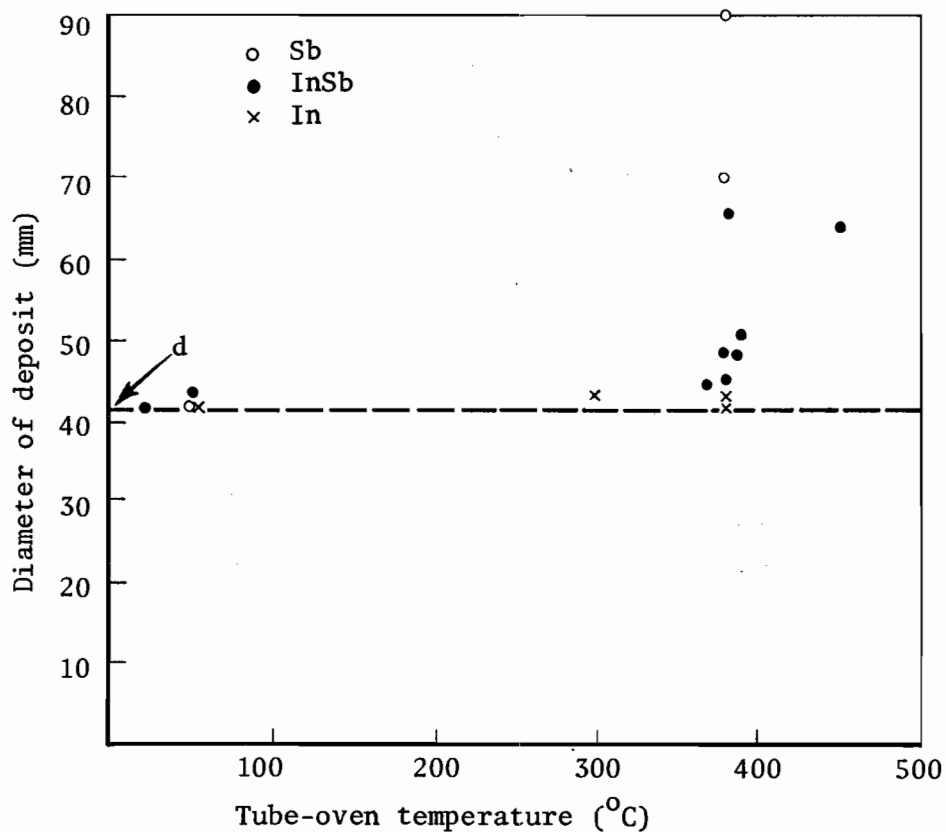

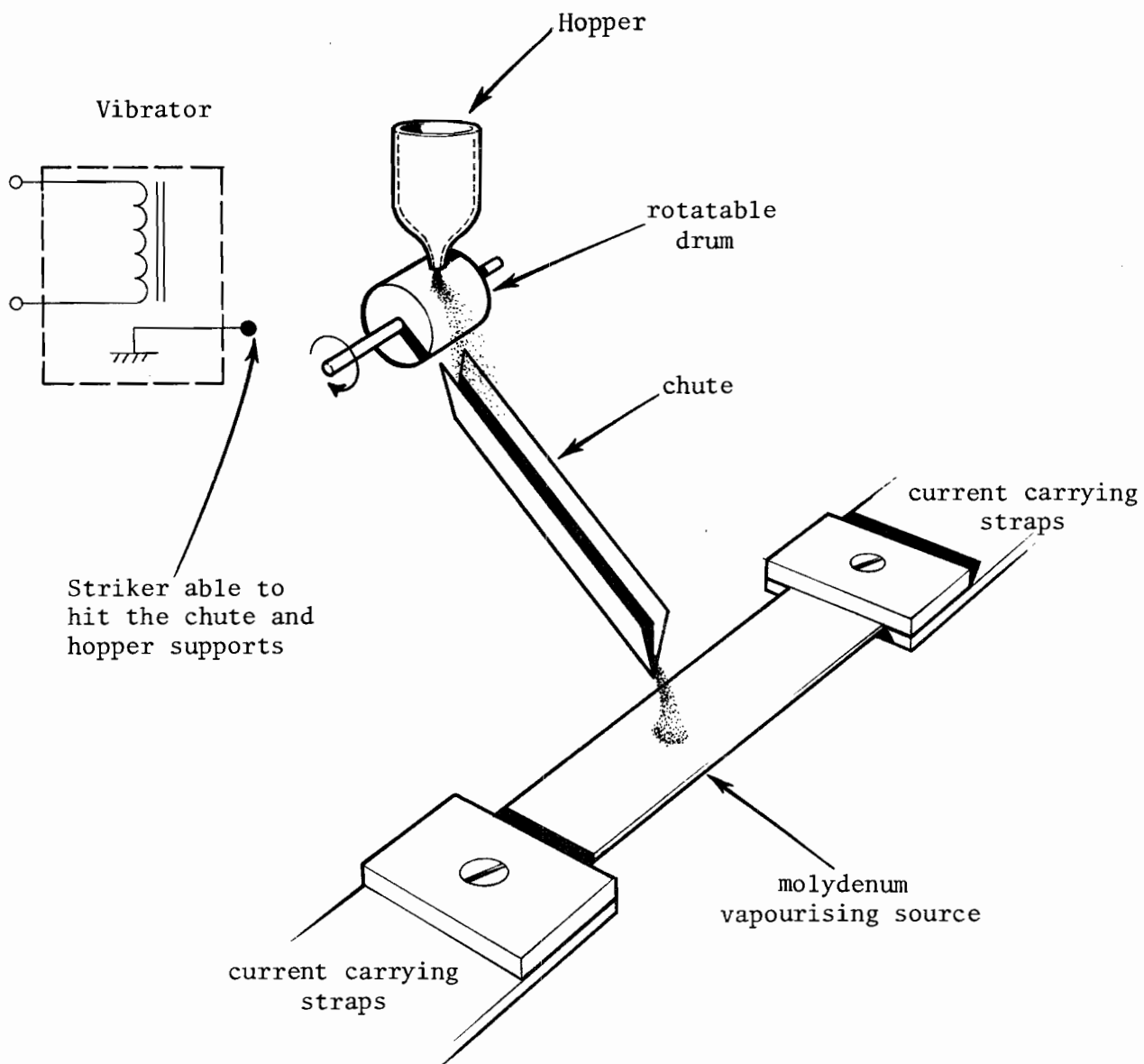Figure 6. The variation of the diameter of the deposit, with tube temperature

Figure 7.   The powder feed mechanism
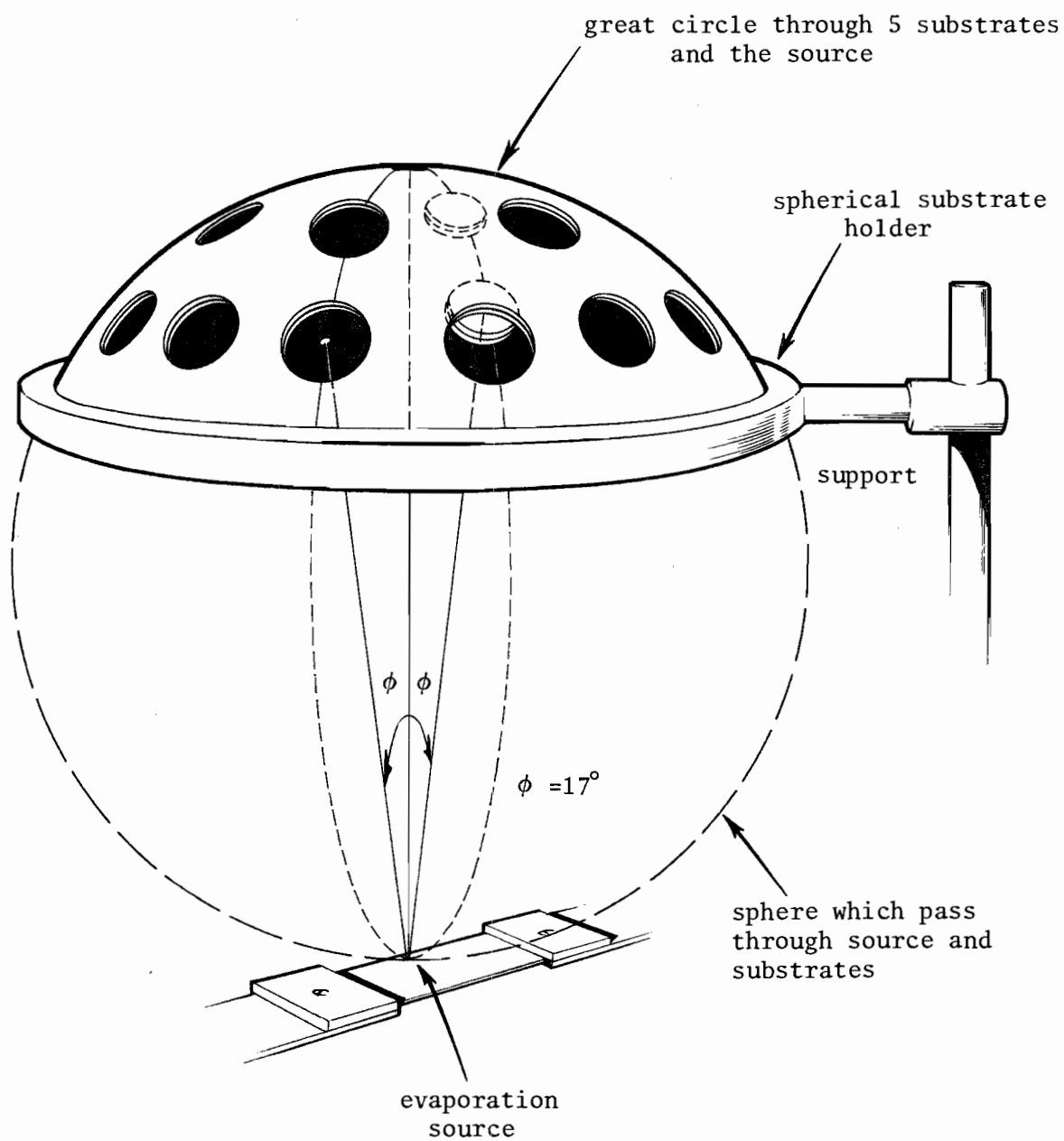
great circle through 5 substrates
and the source

spherical substrate
holder

support

$\phi \quad \phi$

$\phi = 17°$

sphere which pass
through source and
substrates

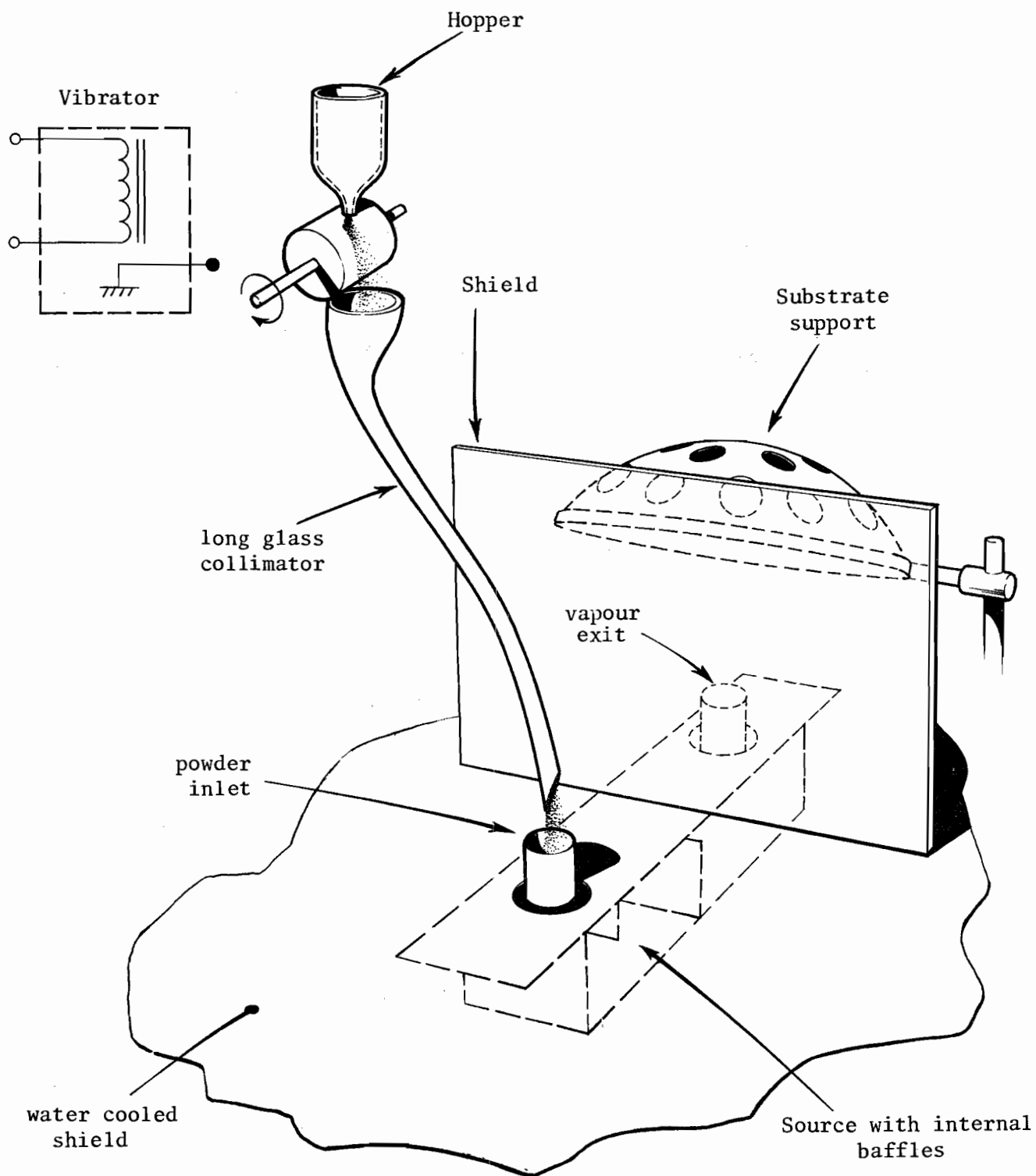evaporation
source

Figure 8.   Placement of source and substrate holder

Figure 9.   Black body source with baffles and collimator

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It has been shown experimentally for deposition rates of practical interest, that the flash evaporation of powdered indium antimonide from a black body source yields a stoichiometric vapour over a wide angular range about the axis of the source. Films deposited by this means have an excellent surface finish and are free from the surface defects often associated with flash evaporation employing other source geometries.